Declaration of Shoichiro Shio

Country of Japan

Prefecture of Kanagawa

City of Yokohama

DECLARATION OF SHOICHIRO SHIO

The undersigned, Shoichiro Shio, states that he is competent to testify and has personal knowledge of the facts set forth below:

1. I have been employed as a research chemist by Shiseido Corporation since 1980.

2. I have worked in the area of nanopowders in general, since 1985, and in the area of titanium nanopowders in particular, since 1985.

3. I earned my Bachelor of Science Degree in Chemistry from Tokyo University of Science, Tokyo, Japan, in 1974, and my Master of Science Degree in Chemistry from Tokyo University of Science, Tokyo, Japan in 1978. I am an inventor of 14 Japanese patents and 2 United States patents.

4. I am an author of 3 peer-reviewed scientific articles in journals including Chemical Communications.

5. I am the primary inventor of the subject matter claimed in U.S. Patent Application Serial No. 10/550,461, ("the '461 application"), which is pending before the USPTO, a patent application assigned to Shiseido Corporation, and which is the subject of this declaration.

6. I carried out an experiment as a comparison to the method of my application. The method and particles were made by a process similar to that disclosed in the patent to Chopin, et al., U.S. Patent Number 6,740,312, ("Chopin"). My reaction conditions were similar to those carried out in the Chopin patent. In my reaction, titanyl sulfate as a titanium source ($TiOSO_4$) (0.1 mol in 100 mL H_2O = 1 M concentration) was heated at 90 °C for 3 hours. The pH was adjusted to 7 with NaOH and the resulting precipitate was filtered. No glycerol, or any alcohol was used.

7. The obtained filtrate was water washed several times, and dried for 12 hours at 105 °C. A sample of the resulting powder is shown in Appendix A attached hereto. It is clear from the photomicrograph that the result of the reaction is a non-spherical agglomerated powder having dimensions in the range of 3 microns, and has low surface area.

8. Example 2 of Chopin involves heating to the boiling point (just below 100 °C) for three hours a titanium oxychloride solution including 10g of anatase TiO_2 seeds, the seeds prepared from a titanyl sulfate solution as detailed at column 5, lines 43-54 of Chopin, adjusting the pH to 9 with NaOH, filtering, washing, and drying the particles for four hours at 150 °C. Glycerol is not used.

9. I believe that the lack of glycerol used in the example of paragraphs 6 and 7, above, as well as in Chopin, leads to non-spherical agglomerated particles with low surface area. I believe that the processes of Chopin do not lead to spherical titanium oxide particles having the characteristics as claimed in the '461 application.

10. Chopin achieves a non-agglomerated state only by the addition of the coating, such as silica or alumina. Without such coating, the powders of Chopin are agglomerated.

Declaration of Shoichiro Shio

11. This declaration is made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon. All statements made are of my own knowledge are true and all statements made on information and belief are believed to be true.

Further Declarant sayeth naught.

Shoichiro Shio

Shoichiro Shio

Signed this 27th of October, 2009.

Application Serial No 10/550,461

Declaration of Shoichiro Shio

Appendix A